



TITLE:

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AUTHOR(S):

Horio, Masao; Takahama, Michihiro

CITATION:

Horio, Masao ...[et al]. Studies on Manufacturing Dissolving Pulp from Bagasse. Bulletin of the Institute for Chemical Research, Kyoto University 1958, 36(5): 172-180

ISSUE DATE:

1958-09-30

URL:

<http://hdl.handle.net/2433/75666>

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Studies on Manufacturing Dissolving Pulp from Bagasse

Masao HORIO and Michihiro TAKAHAMA

(Horio Laboratory)

Received August 19, 1958

The fundamental researches and a semi-commercial scale tests by the prehydrolysis sulfate process were carried out on manufacturing dissolving pulp from the Indian bagasse. The prehydrolysis at 170°C for 30 min. with an addition of 1% H₂SO₄ followed by sulfate cooking with 25% cooking liquor at 170°C for 30 min. is optimum. The total yield is about 20%. The fiber length of the bagasse in the dissolving pulp is about 1.69 mm. in average and contains a very short fiber of the length of 1—1.5 mm. The bleaching of the pulp is rather easy, by an ordinary five stage bleaching process, the whiteness can be reached up to 85—90%. As far as the chemical composition is concerned, the dissolving pulp made from the Indian bagasse is satisfactory except rather high content of ash. Because of the short fiber length, the viscose process is difficult unless the bagasse pulp is blended with the other pulp having long fiber length. The industrialization of dissolving pulp production from the Indian bagasse has a number of problem and at present situation, it would be impossible in a commercial scale plant.

I. INTRODUCTION

The utilization of the bagasse, an waste material from cane sugar manufacturing factories, is an important industrial problem especially in India. However, mainly because of the bulkiness, the difficulties on transportation and storage have been blocking the way for the industrial utilization of the bagasse. Under the present situations, the most part of the bagasse is used as a fuel in those cane sugar factories, and only a small portion is utilized as a material for manufacturing paper.

The present report deals with the possibility of manufacturing dissolving pulp from the Indian bagasse in a commercial scale. A number of researches have been carried out on the manufacturing paper pulp from the bagasse, but only a few papers have been published on manufacturing dissolving pulp from the bagasse. Recently, George Jayme and Karl-Heinz Rosenstock¹⁾ have made an extensive studies on the manufacturing dissolving pulp from the Peruvian bagasse. In the present studies, in order to obtain more realistic data on the problem, we have carried out a series of researches from the pulp making processes throughout the viscose rayon spinning processes using a semi-commercial plant. The data which we have obtained in our previous researches²⁾ on manufacturing dissolving pulp from the Indian bamboo were effectively applied in the course of the present studies.

II. BAGASSE, THE RAW MATERIAL

The chemical analysis of the Indian bagasse including its pith used in the

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present experiments gave the following composition as shown in Table 1.

As in the case of the Indian bamboo, the Indian bagasse shown in a high ash content. The result of chemical analysis of the ash is shown in Table 2.

The composition of ash was similar with that of the Indian bamboo. Since

Table 1. Chemical analysis of the Indian bagasse.

Components	Inclusion ratio(%)
Water	1.40
Ash	2.34
Alcohol-benzen extracts	4.52
Pentosan	17.70
Lignin	21.15
Total cellulose	43.40
	$\left\{ \begin{array}{l} \alpha \text{ Cellulose } 53.55\% \\ \beta \text{ Cellulose } 21.41\% \\ \gamma \text{ Cellulose } 25.09\% \end{array} \right.$
Water soluble substance	10.38

Table 2. Chemical analysis of the ash.

Components	Content(%)
SiO ₂	67.23
Fe ₂ O ₃	0.59
CaO	7.41
MgO	4.01
K ₂ O	10.75
Na ₂ O	1.1
Oxides of Al, P, Mn	Slight
Oxides of Zn, B, Pb.	Trace

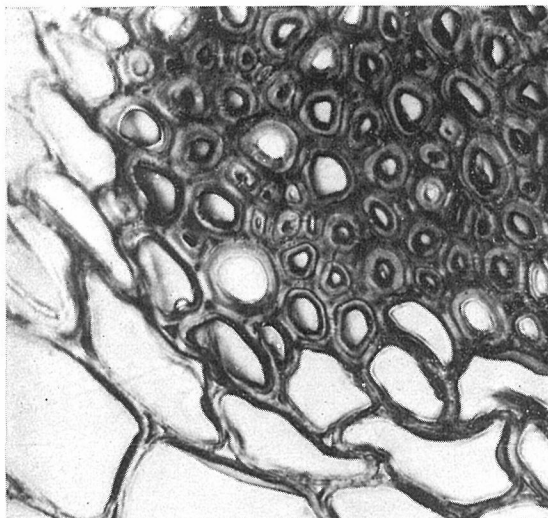


Fig 1. Cross Section of the Indian bagasse.

the pith of the bagasse has a very high content of starch and consumes the cooking liquor considerably, it is desirable to separate the pith before cooking process, unless the recovery of furfural or glucose from prehydrolysis waste liquor is designed in the pulping plant.

Figure 1 shows the microscopic structure of a section of the Indian bagasse. As in the case of the Indian bamboo, the fiber cells are surrounded by parenchyma cells.

III. PREHYDROLYSIS

In order to remove pentosan and the other polysaccharides, we have applied the prehydrolysis process before the sulfate cooking process. Based on our data for the case of the Indian bamboo, we have chosen the following conditions for the prehydrolysis process, at 170°C both steam-prehydrolysis and H₂SO₄-prehydrolysis were carried out. The results are shown in Table 3 and Fig. 2.

As seen in Table 3 and Fig. 2, the prehydrolysis with an addition of H₂SO₄ is very effective for removal of pentosan. The yield of the prehydrolysis was about 65%, which was about 10~15% less than that for the case of the Indian bamboo. This lower yield is probably due to the high content of water soluble components in the pith of bagasse.

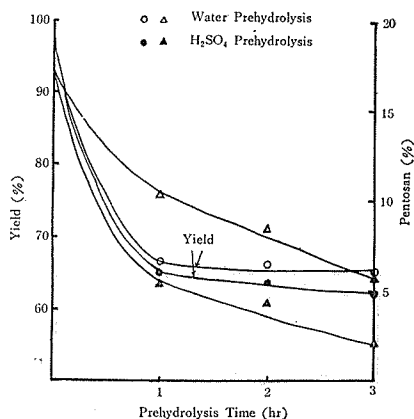


Fig. 2. Prehydrolysis of bagasse.

Table 3. Removal of pentosan in prehydrolysis.

Prehydrolysis medium	Prehydrolysis temperature (°C)	Prehydrolysis time (hr)	Yield %	Pentosan %
Steam	170	1	66.67	10.41
		2	66.10	8.51
		3	65.02	5.94
H ₂ SO ₄ 1%	170	1	65.15	5.61
		2	63.60	4.42
		3	62.03	2.19

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The bagasse occupies about twice the volume of wood of the same weight. But by the prehydrolysis process, the volume is reduced to less than one half of the original volume, whereas the Indian bamboo keeps the original volume even after the prehydrolysis process.

IV. SULFATE COOKING

The sulfate cooking of the bagasse was carried out on the sample which have been treated by the steam-prehydrolysis or the H_2SO_4 -prehydrolysis, as described above. The results are shown in Table 4 and in Figs. 3 and 4.

Table 4. Sulfate cooking of prehydrolyzed bagasse.

Prehydrolysis	Conditions of sulfate cooking					Analysis data of unbleached pulp			
	Cooking agent added for bagasse (%)	Temp. (°C)	Time (hr)	Yield (%)	Residue (%)	P.N.	Pentosan	Cellulose (%)	D.P.
Medium: water			1	25.8	0.52	3.71	1.76	95.44	700
Temp.: 170°C	25.0	165	2	24.7	0.36	3.63	1.63	95.20	590
Time: 1hr			3	24.2	0.52	3.37	1.54	94.07	470
Pentosan content of prohydrolyzed	20.0		1	22.4	1.79	4.05	1.68	96.13	750
Bagasse: 9.36%	17.5	165	1	21.1	3.28	4.90	1.57	96.03	770
Yield: 69.44%	15.0		1	22.5	2.98	6.79	1.73	96.54	904
Medium: H_2SO_4 1%			1	21.9	1.35	4.77	1.31	97.47	600
Temp: 170°C	25	165	2	20.5	1.70	3.69	1.23	93.97	490
Time: 1hr			3	22.3	0.70	3.09	0.99	92.18	460
Pentosan content of prehydrolyzed	20		1	20.1	4.11	4.50	1.47	94.73	660
Bagasse: 4.80%	17.5	165	1	20.5	3.80	5.04	1.03	94.45	710
Yield: 60.52%	15.0		1	19.4	4.76	6.81	1.09	95.97	730

The compositions of the cooking liquor used in this sulfate cooking was the followings :

NaOH	155.04 g/l
Na ₂ S	67.68 g/l
Total alkali	222.72 g/l
Sulfidity	30.41 %
Liquor ratio	2.5

The low permanganate number, as seen in Table 4, shows that the liquor in the bagasse is easily removed by the sulfate cooking process. One hour of cooking with 25% cooking liquor would be suitable from the data of yield and residue shown in Table 4.

As far as the yield of prehydrolysis is concerned, the steamprehydrolysis giv-

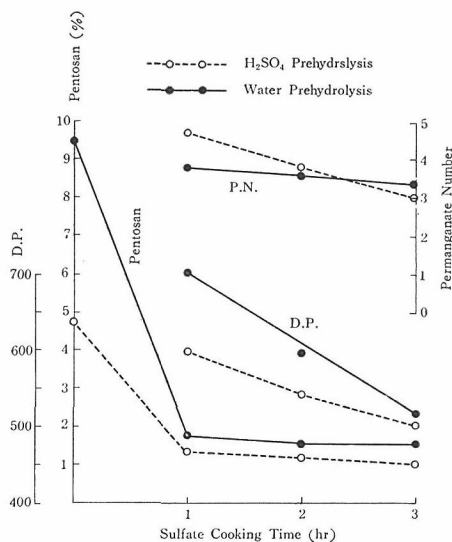


Fig. 3. Sulfate cooking of bagasse.

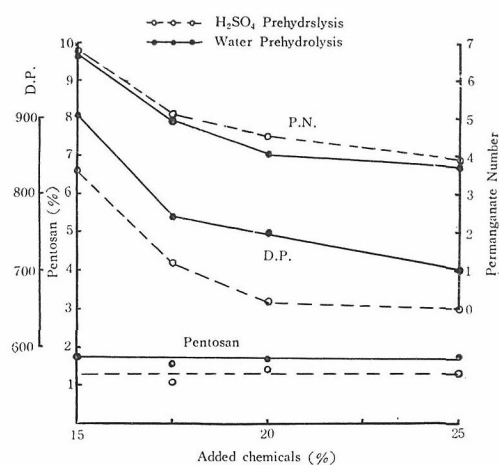


Fig. 4. Sulfate cooking of bagasse.

ing 24-25% yield is better than the H₂SO₄-prehydrolysis giving 20-22% yield, however, the pentosan content in the case of steam-prehydrolysis is too high for the viscose process. Therefore, in spite of the low yield, it would be the best way to choose the H₂SO₄-prehydrolysis and to consider some utilization of polysaccharide extracted in the process of prehydrolysis.

Figs. 3 and 4 show microscopic pictures of sections of pulp dyed with 0.05% Malachite Green; Fig. 5 for the pulp prepared by means of the steam-prehydrolysis and Fig. 6 for that by the H₂SO₄-prehydrolysis. Fig. 7 is the microscopic picture of bagasse fibers in pulp prepared by means of the steam-prehydrolysis, and Fig. 8 is that of pulp by the H₂SO₄-prehydrolysis. The parenchyma cell are

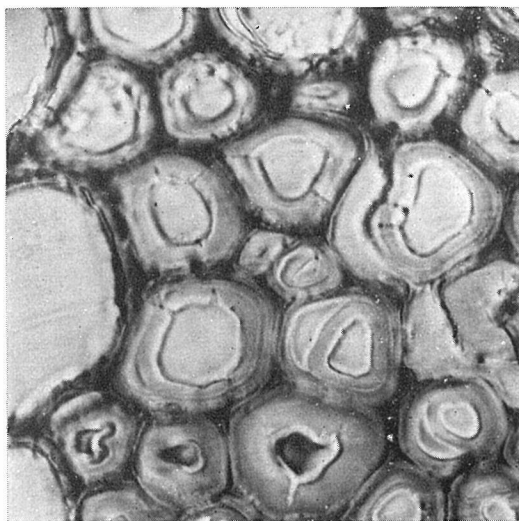
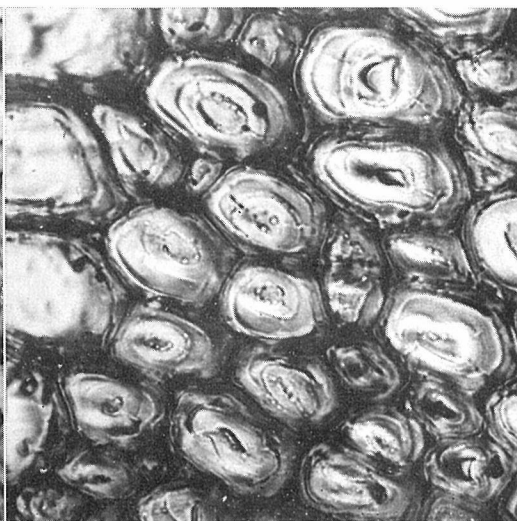


Fig. 5. Microscopic cross section of Indian bagasse pulp (prehydrolysis with water).

Fig. 6. Microscopic cross section of Indian bagasse pulp (prehydrolysis with H₂SO₄).

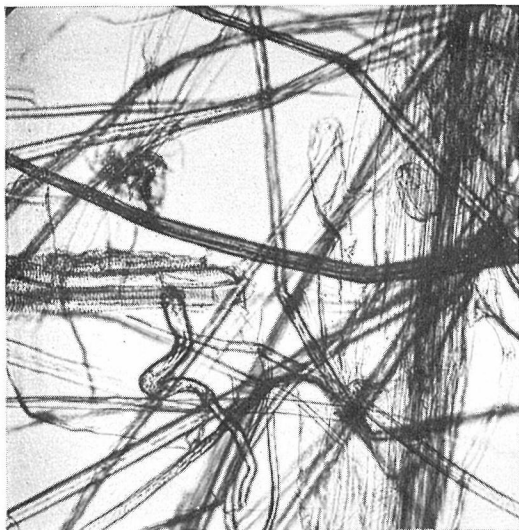


Fig. 7. Microscopic photo of Indian bagasse pulp (prehydrolysis with water).



Fig. 8. Microscopic photo of Indian bagasse pulp (prehydrolysis with H_2SO_4).

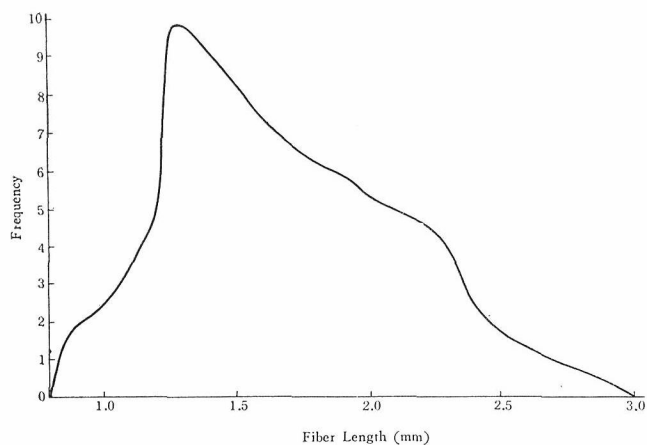


Fig. 9. Distribution of fiber length.

seen in both Figs. 7 and 8, but, as seen these pictures, the parenchyma cells are reduced in the case of the H_2SO_4 prehydrolysis.

The average length of fibers in the bagasse pulp thus prepared was about 1.69 mm, and the distribution of fiber length is shown in Fig. 9.

The freeness was 5.0 sec. for the unbleached bagasse and 6.3 sec. for the bleached bagasse pulp; these figures were considerably higher than that the Indian bamboo pulp which were 4.5 sec. for the unbleached one and 4.7 sec. for the bleached one.

V. EXPERIMENTS IN THE SEMI-COMMERCIAL PLANT

The manufacturing of dissolving pulp and the spinning of viscose rayon

Table 5. Semi-commercial tests.

Experimental No.		1	2	3
Prehydrolysis	Medium	Steam	H ₂ SO ₄ 1%	H ₂ SO ₄ 1%
	Temperature (°C)	165	170	170
	Time required to reach the temperature (hr.)	1	1	1
	Time at the temperature (hr)	1	0.5	0.5
	Total Time	2	1.5	1.5
Sulfate cooking	Amount added (%)	25	25	25
	Liquor ratio (Kg/l)	2.5	2.5	2.5
	Temperature	165	170	170
	Time required to reach the temperature (hr)	2.0	1.5	1.5
	Time at the temperature (hr)	1.5	1.0	0.5
	Total time (hr)	3.5	2.5	2.0
Unbleached pulp	Yield (%)	23.42	20.32	21.3
	Residue (%)	1.34	0.35	0.1
	Permanganate number	3.66	2.73	3.32
	Pentosan (%)	2.71	1.73	1.45
	α -Cellulous (%)	96.1	95.9	95.9
	Degree of polymerization	840	700	935
Bleached pulp	α -Cellulose (%)	92.4	93.6	93.2
	β -Cellulose (%)	3.2	3.0	4.5
	γ -Cellulose (%)	1.6	1.9	1.2
	Ash (%)	1.34	0.57	1.18
	Alcohol benzen extracts (%)	0.35	0.15	0.15
	Pentosan (%)	3.02	1.75	1.55
	Degree of polymerization	580	435	735
	Whiteness (%)	85.7	89.2	88.7
Viscose tests	KW value	2820	1986	1530
	Aging conditions	18°C, 6hr	18°C, 4hr	23°C, 18hr
	Viscosity (Sec)	36.2	34.4	55.4

Table 6. Experimental conditions of multistage bleaching of the Indian bagasse.

Stage	Consistency of pulp(%)	Added chemicals	Temperature (°C)	Time (Min.)	pH
1	3	P.N. \times 1.355 \times 1.4 \times 0.8 \times 0.8	30	60	10
2		P.N. \times 1.355 \times 1.4 \times 0.8 \times 0.2	20	30	1.5
3		2	65	30	—
4		P.N. \times 1.355 \times 1.4 \times 0.12	40	60	10
5		P.N. \times 1.355 \times 1.4 \times 0.08	40	60	10

filament were carried out in a semi-commercial plant, based on the data obtained in our fundamental studies described above. Because of the bulkiness of the bagasse, we have carried out two batches of cooking under the same conditions. The conditions of cooking and the analytical data are shown in Table 5.

The conditions used in the bleaching process is shown in Table 6.

The bagasse pulp thus prepared was easier to bleach compared with the Indian bamboo pulp. The whiteness could reach up to 85~90% by means of ordinary five stage bleaching process. However, it is still desirable to apply the ClO_2 treatment for these sulfate pulp.

The pentosan content in the bleached bagasse pulp was about 3% in the case of the steam-prehydrolysis (165°C, 1hr.) and about 1.5% in the case of the H_2SO_4 prehydrolysis (170°C, 30 min.).

Therefore, as a conclusion, the optimum conditions are the prehydrolysis with an addition of 1% H_2SO_4 for 30 min. at 170°C and then the sulfate cooking at 170°C for 30 min., in this case the final yield being about 20%. The pulp thus obtained showed 93% of α -cellulose and more than 0.5% of ash content. The main source of ash seemed to be the parenchyma cell. Hence, in order to reduce this ash content, it is necessary to carry out through washing to remove parenchyma. Table 7 shows the main components of ash in each stage of the pulping process. The abnormally high Fe_2O_3 content in the bleached pulp is mainly due to the imperfection of the experimental apparatus. In the case of sample No.2 in Table 5, the ash content in the bleached pulp could be reduced down to 0.57%, which corresponds to that of the Indian bamboo pulp.

Table 7. Main components of ash in each stage of the pulping process.

Item	$\text{SiO}_2(\%)$	$\text{Fe}_2\text{O}_3(\%)$	$\text{CaO}(\%)$	$\text{MgO}(\%)$	K^+		Na^+	
					mg	mg	mg	mg
Bagasse	67.23	0.59	7.41	4.01	10.75	152.9	11	152.9
Prehydrolysis liquor	14.41	13.79	10.19	5.49	4.25	90.30	5.0	90.30
Black liquor	1.69	8.72	—	—	0.165	129.0	43.0	129.0
Unbleached pulp	33.86	6.21	20.65	11.56	0.965	119.39	1.95	119.39
Bleached pulp	31.21	15.71	33.61	8.14	0.325	130.55	0.51	130.55

VI. VISCOSE PROCESS

As shown in Fig 9, the fiber length of the bagasse pulp was 1.69 mm in average and contained a considerable portion of shorter length 1~1.5 mm. Due to this short fiber length, the filterability of this pulp was very poor, and the mechanical strength of the pulp was not good enough for the viscose processes. During the alkali steeping, the bagasse pulp was easily broken by a slight agitation, and the compression process had to be carried out with great care. The disintegration of the alkali cellulose could be done in a continuous disintegrator but not by an Werner type disintegrator. The xanthation of the alkali cellulose was quite difficult due to the formation of xanthate mass. The viscose

thus prepared showed a high free fiber content. These situations are similar with the case of beech pulp which is not suitable for dissolving pulp unless blended with pine pulp.

As far as the analytical data is concerned, the bagasse pulp gives good figures for a dissolving pulp, but, because of the short fiber length, the bagasse pulp is not suitable for the viscose rayon process.

VII. CONCLUSION

Based on our studies reported in the present paper, we conclude the following points for manufacturing dissolving pulp from the Indian bagasse :

(1) The bagasse, as far as the price and the sample supply is concerned, would be a good raw material pulp industry in India.

(2) For making dissolving pulp from the Indian bagasse, it is highly desirable to separate the pith before the processes.

(3) Because of the bulkiness, the capacity of pulp making apparatus should be specially considered. On the H_2SO_4 -prehydrolysis, it requires the prehydrolysis liquor ratio of 10. On the steam-prehydrolysis, about 3 hours cycle is required. All of these factors are the main disadvantages in the utilization of the Indian bagasse in pulp industry. And also, special consideration must be given to the fact that the volume of the Indian bagasse is, after the prehydrolysis process, reduced to one half of the original volume. Although it is desirable to use separate apparatus for the prehydrolysis and the sulfate cooking, in a commercial scale, a continuous cooking process should be designed specially for the Indian bagasse.

(4) During the prehydrolysis process, about 35~40% of the original bagasse is dissolved out. This soluble part of the bagasse consists of pentose and the other polysaccharides. Since the final yield of the bleached pulp is only about 20%, the recovery of glucose or furfural from the waste prehydrolysis liquor should be considered as a by-product of the dissolving pulp plant.

(5) Since the fiber length of the Indian bagasse in the dissolving pulp is so short that the viscose processes are very difficult unless a special technique for handling the bagasse pulp is developed. 30% of blending bagasse pulp into the Indian bamboo pulp would be the maximum ratio for the viscose process.

(6) Because of the different cooking conditions, it would be rather difficult to handle the bagasse in the same plant handling the Indian bamboo.

Considering the facts described above, although it is possible to make a dissolving pulp from the Indian bagasse in a laboratory scale, the industrialization of the bagasse dissolving pulp would be very difficult. For a paper-pulp, bagasse can give the yield of about 40%, and as a fuel, bagasse has about 4380 cal.

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